



## Original article

# Permeability test and fuzzy orthogonal analysis of hydrogenated nitrile O-ring

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## ABSTRACT

In the high temperature, high pressure and high corrosive environment of the oil and gas drilling downhole, the weatherability of rubber sealing material has a great influence on the production safety. In order to study the important degree of every key environmental factor in downhole influencing the sealing performance of rubber sealing material, a new device of simulating downhole environment is designed to test the permeability of O-ring. The sample is hydrogenated nitrile O-ring and orthogonal experiment method is used to do nine tests by getting three levels from temperature, pressure and CO<sub>2</sub> volume fraction. Test adopts fuzzy orthogonal method to analyze the main effects and the interaction between two factors, taking tensile strength, diameter variety rate and pH value of indicator as evaluation index. The results show that: the environmental factor influencing the sealing performance of hydrogenated nitrile O-ring from high to low by turns is temperature, pressure and CO<sub>2</sub> volume fraction, while the interaction between temperature and pressure is the most significant. It provides a new way to study the influence of downhole complex environment on the performance of rubber sealing material. Moreover, the results have important reference value to further study the failure mechanism of rubber sealing ring in many environmental factors and the rational use in engineering.

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Rubber seal products have been widely used in oil and gas production, such as the sealing ring of marine riser, the rubber of packer and the rubber core of blowout preventer, which are convenient in manufacture, satisfactory in sealing performance and inexpensive [1]. With the development of petroleum industry and the drilling depth increasing, the demands for rubber sealing materials are becoming more and more critical. Fluorine rubber and nitrile rubber are commonly used in petroleum drilling engineering [2,3]. Hydrogenated nitrile rubber is a new variety after nitrile rubber, which has more excellent performance in every respect. Hydrogenated nitrile rubber was developed in 1980's, which has good resistance to high

temperature and high pressure besides the excellent oil and chemical resistance as nitrile rubber. Moreover, the tensile strength performance of hydrogenated nitrile rubber is also excellent because of the tensile crystallization [4]. When the drilling or oil-gas production is carried out, rubber sealing material is usually in a high temperature, high pressure and high corrosive media environment, which are the main factors that make rubber sealing material become aging and deform. It is clear that the downhole work environment has great effect on the sealing performance of rubber [5–8]. So, it has important significance to research the sealing performance and the main failure reason of rubber seal ring in work environment.

At present, the researches about sealing performance of rubber material have many achievements. Rodriguez P L [9] studied the influence results about different factors to the leakage model of rubber O-ring in the space environment; Wang Guangzhen [10] studied the influence of compression ratio, temperature and load attenuation on sealing performance of rubber O-ring; Peter Warren [11] studied the sealing capability of rubber O-ring in low temperature; Zhang Mei [12] studied the

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sealing capability of common rubber O-ring in super-high pressure environment; Junichiro Yamabe [13] studied the failure behavior of rubber O-ring under cyclic exposure to high-pressure hydrogen gas; Zeng Dezhi [14] studied the corrosion resistance to acid medium of fluorine rubber O-ring in compressive stress state. There are also other studies about the seal test of the rubber ring [15–19]. But, there isn't a comprehensive study about the importance of various environmental factors affecting the performance of the rubber seal ring.

The orthogonal test with hydrogenated nitrile O-ring is carried out in simulation downhole environment of oil and gas exploitation to test the effects about temperature, pressure and concentration of corrosion medium to O-ring's sealing performance. Then, fuzzy orthogonal method is used to deal with the test data and analyze the main factors and the interaction between two factors of hydrogenated nitrile O-ring seal failure. This paper provides a new way to study the influence of downhole complex environment on the performance of rubber sealing material. Moreover, the results have important reference value to further study the failure mechanism of rubber sealing ring in many environmental factors and the rational use in engineering.

## 1. Test part

### 1.1. Specimens and environment

27 pieces of hydrogenated nitrile O-ring specimens is used in test, the specification of which is  $\Phi 75 \text{ mm} \times 3.6 \text{ mm}$ . Each has 3 parallel tests and a total of 9 groups. The surface of specimens are smooth and not damaged, which is consistent with the GB/T3452.2 about appearance quality inspection standard of rubber O-ring.

In order to test the performance of hydrogenated nitrile rubber in downhole environment of highly corrosive gas, the sample of formation water and environment data in a drilling platform are collected to make sure permeability test of hydrogenated nitrile O-ring go on wheels. The component of water sample are shown in Table 1 and the test environment is set to: the temperatures are 80 °C, 100 °C and 120 °C, the pressures are 15 MP, 20 MP and 25 MP, the volume fractions of CO<sub>2</sub> gas (according to the content of water to simulate corrosion environment) are 5%, 10% and 15%.

### 1.2. Test equipment and instrument

High temperature and high pressure reactor is used as environment control device of the experiment. The maximum sealing pressure is 60 MPa, the maximum operating temperature is 180 °C and the volume is 7 L. As show in Fig. 1, pressing the gas into the reaction kettle realizes the purpose of increasing pressure and simulating corrosion environment. Besides, the heater around the kettle can heat the kettle and the temperature sensor and pressure gauge in the cover of reaction kettle can observe and control environmental variations of the kettle. After the experiment, the high-pressure air can be exhausted from air outlet. So, the parameters setting of different temperatures, pressures, concentration of corrosion medium in downhole

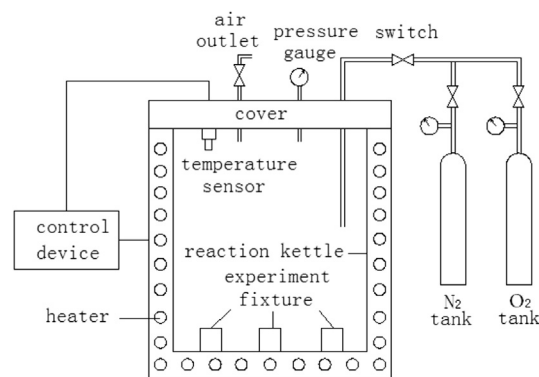


Fig. 1. Illustrative diagram of experiment device.

environment can be realized to finish the permeability test of hydrogenated nitrile O-ring [20].

In order to finish sealing performance test of hydrogenated nitrile O-ring in high temperature, high pressure and high corrosive environment, the experiment fixture is designed. As shown in Fig. 2, rubber O-ring is inserted into the groove of the container cover and Na(OH) indicator is installed on the container base. The venthole is used to drain out the excess gas from the container when pressing in the container cover and timely detect the pH value of indicator in the container after experiment. The metal C-ring has a sealing function between plate and container cover to prevent gas from entering container. The vent groove is conducive to display agent and permeate gas having a better reaction. According to the O-ring's size and 15% compression ratio of initial assembly, the mating dimensions of container cover and base are designed. Moreover, each of the components is manufactured according to the size of design in the tolerance range to ensure sealing.

The tensile property of O-ring after experiment is tested by using the JWL-2500N type electronic tensile testing machine, the display agent pH value of indicator in fixture before and after the experiment is tested by PHS-2F desktop pH meter and the O-ring's inner and outer diameters are tested by the vernier caliper of precision 0.01 mm.

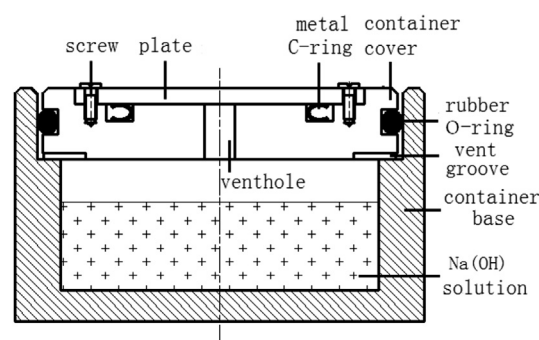


Fig. 2. Schematic diagram of experiment fixture.

Table 1  
The main ion content and physical parameter of sampling strata water.

Main ion content/(mg L <sup>-1</sup> )							Density/(g cm <sup>-3</sup> )	Total salinity/(mg L <sup>-1</sup> )	pH
K <sup>+</sup>	Na <sup>+</sup>	Ma <sup>2+</sup>	Ca <sup>2+</sup>	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	HCO <sub>3</sub> <sup>-</sup>			
30.44	1081.51	7.84	24.98	763.02	36.21	101.87	0.993 g/L	2045.87	7.63

### 1.3. Test methods and steps

This test uses orthogonal experiment method to divide the environmental parameters of different values (temperature, pressure, volume fraction of CO<sub>2</sub>) into groups. In this way, the number of test can be reduced as much as possible in the premise of not affecting the test results to save time and cost. Besides, setting a parallel sample to calculate the average value can reduce the test error. Test fixture is put into the reaction kettle and the simulation parameters environment is set. After 24 h, the sample is removed to test the inner and outer diameter vale, the tensile strength and the display agent pH values in the base container of O-ring. Then, the fuzzy orthogonal method is used to analyze the effect of environment factors to the evaluation index and find the key factors in environment variables.

Steps: (1) Dividing the 27 pieces of hydrogenated nitrile O-ring into 9 groups(each group have three pieces parallel sample) and measuring the average radial diameter  $d$ . (2) Taking equal Na (OH) indicator of pH 10.12 into the every sealing base and completing the fixture assembly of bearing state. (3) Inverting the fixture into a corresponding position of reaction kettle and sealing reaction kettle. (4) Putting N<sub>2</sub> into the reactor for a period of time to discharge O<sub>2</sub> and pressuring N<sub>2</sub> and CO<sub>2</sub> into the dynamic reactor according to respective volume fraction until the pressure of reactor reaches the specified value. (5) Heating the reaction kettle and preserving heat when reaching the specified numeric. (6) Putting out the sample after 24 h in the specified environment and testing the radial diameter, tensile strength and the pH value in the container base of each sample. (7) Recording data and completing data analysis.

### 1.4. Test result and data

As can be seen from Fig. 3, a portion of the sample after test has varying degrees of bubbling and deformation, which has a greater impact on the sealing properties.

The test data can be recorded as Table 2 through the measurements of the detection device. The sealing properties of hydrogenated nitrile rubber O-ring decrease with the increase of temperature, pressure and volume fraction of CO<sub>2</sub>, namely showing that the permeability resistance weakened. The difference value (before and after the experiment) divided by the rubber O-ring's diameter before the experiment, which is the diameter change rate. The test data need to be processed to get the impact result of environment factors to evaluation index and find out the key factors.

## 2. Analysis of test results

### 2.1. Determine the index and object sets

Putting evaluation index set: tensile strength  $Y_1$ , diameter change rate  $Y_2$  and pH value  $Y_3$  as the evaluation index set, namely  $U = \{Y_1, Y_2, Y_3\}$  [21].

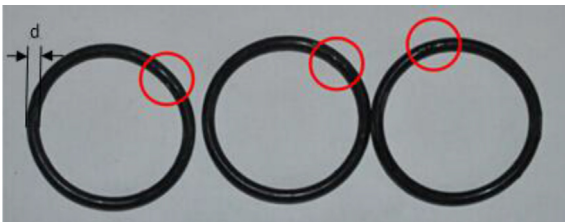


Fig. 3. The sample after test.

Table 2

The orthogonal test design and test results.

Number	Temperature/ °C	Pressure/ MP	Volume fraction(CO <sub>2</sub> )/ %	Tensile strength/ MP	Diameter change rate/%	pH value
1	80	15	5	24.6	2.3	9.85
2	80	20	10	24.3	2.7	9.56
3	80	25	15	24.3	2.6	9.27
4	100	15	10	24.1	3.4	9.11
5	100	20	15	23.7	3.7	8.96
6	100	25	5	23.5	3.8	8.45
7	120	15	15	23.2	5.1	8.23
8	120	20	5	23.3	5.3	8.06
9	120	25	10	22.9	5.2	7.87

Putting 9 groups orthogonal test as the evaluation objects set, namely  $D = \{d_1, d_2, \dots, d_9\}$ .

### 2.2. Establish membership functions

Establishing the membership functions of evaluation index set  $U$  to evaluation set  $V$  ( $V$  is used to describe the extent of the evaluation index set according with the evaluation criteria) and making the size of membership values calculated according to membership function to adapt the importance of related index in comprehensive evaluation. Membership function is a monotonic function and the degree of membership  $r_{mn}$  is between 0 and 1 ( $m = 1, 2, 3; n = 1, 2, \dots, 9$ ). If the value of evaluation index is larger and the performance of rubber O-ring is better after experiment, evaluation index should take the larger value and vice versa. The membership function of membership degree as flows:

$$Y_{1n} \text{ takes the larger value, so the membership function}$$

$$r_{1n} = \frac{(Y_{1n} - Y_{1nmin})}{(Y_{1nmax} - Y_{1nmin})} \quad (1)$$

$$Y_{2n} \text{ takes the smaller value, so the membership function}$$

$$r_{2n} = \frac{(Y_{2nmax} - Y_{2n})}{(Y_{2nmax} - Y_{2nmin})} \quad (2)$$

$$Y_{3n} \text{ takes the larger value, so the membership function}$$

$$r_{3n} = \frac{(Y_{3n} - Y_{3nmin})}{(Y_{3nmax} - Y_{3nmin})} \quad (3)$$

The membership degree of single index  $Y_{1n}$ ,  $Y_{2n}$  and  $Y_{3n}$  as shown in Table 3. The fuzzy relation matrix  $R$  composited by membership value is:

$$R = \begin{bmatrix} r_{11} & r_{12} & \cdots & r_{1n} \\ r_{21} & r_{22} & \cdots & r_{2n} \\ \vdots & \vdots & \ddots & \vdots \\ r_{m1} & r_{m2} & \cdots & r_{mn} \end{bmatrix} = (r_{mn})_{m \times n} \quad (4)$$

### 2.3. Determine the weight distribution set $A$

$A$  is a fuzzy subset about evaluation index set  $U$ , namely the weight distribution set, which reflects the importance of every index. In this test, the fuzzy subset is  $A = \{0.2, 0.3, 0.5\}$ . Basis for

**Table 3**

The value of single subordinate degree and fuzzy comprehensive evaluation.

Number	Temperature/°C	Pressure/MP	Volume fraction(CO <sub>2</sub> )/%	Tensile strength/MP	Diameter change rate/%	pH value	Comprehensive evaluation value(b <sub>n</sub> )
1	80	15	5	1.000	1.000	1.000	1.000
2	80	20	10	0.824	0.867	0.854	0.852
3	80	25	15	0.824	0.900	0.707	0.788
4	100	15	10	0.706	0.633	0.626	0.644
5	100	20	15	0.471	0.533	0.551	0.510
6	100	25	5	0.353	0.500	0.293	0.367
7	120	15	15	0.176	0.067	0.182	0.146
8	120	20	5	0.253	0.000	0.094	0.098
9	120	25	10	0.000	0.033	0.000	0.010

deciding: PH value is the main index to evaluate the sealing properties of hydrogenated nitrile O-ring in the permeability test; Diameter change rate is used to measure the deformation degree of O-ring and has a larger contact performance with seal performance of O-ring; The tensile strength is used to measure the physical properties of O-ring and has a weaker effect to the sealing performance of O-ring in the short-term test.

#### 2.4. Calculation of the fuzzy comprehensive evaluation value

A fuzzy subset B is introduced(called the evaluation grade) under the evaluation set V, namely  $B = \{b_1, b_2, \dots, b_n\}$ , which can be got by the fuzzy transform of fuzzy matrix R and the weight distribution A.

$$B = A \cdot R \quad (5)$$

There are many methods of fuzzy arithmetic. This paper use arithmetic operators  $M(\cdot, +)$  to make a fuzzy transformation to  $B = A \cdot R$ . The degree membership  $b_n$  of comprehensive evaluation fuzzy subset B can be get by this way. The fuzzy comprehensive evaluation as shown in Table 3.

#### 2.5. Analysis of main effect

The main effect factors are the most important factor influencing sealing performance of O-ring in all environmental factors. Fuzzy analysis method is used to make a comparative analysis of the maximum value of fuzzy sub in various factors to determine the main factor.

The environmental factors of permeability test is set to  $C_i$  about hydrogenated nitrile O-ring. The discourse domain is  $X_i$  about  $C_i$ , namely  $X_i = \{C_{ij}\}$ . Among:  $i = 1, 2, 3$ (environmental factors);  $j = 1, 2, 3$ (the number of levels about each factors).

In order to compare for each level, the total fuzzy comprehensive evaluation of  $C_{ij}$  are normalized and get  $(\sum b_{ij})'$ . It

indicates the influence of the level on fuzzy comprehensive evaluation value and the result as seen in Table 4.

Because  $(\sum b_{ij})'$  on behalf of the membership grade of  $C_i$  in  $C_{ij} \in X_i$ , namely the degree of  $C_{ij}$  belongs to the fuzzy set  $C_i$ ,  $(\sum b_{ij})'$  can be expressed as a fuzzy subset about  $X_i$ . There are:

$$\begin{cases} C_1 = [(\sum b_{11})', (\sum b_{12})', (\sum b_{13})'] \\ C_2 = [(\sum b_{21})', (\sum b_{22})', (\sum b_{23})'] \\ C_3 = [(\sum b_{31})', (\sum b_{32})', (\sum b_{33})'] \end{cases} \quad (6)$$

The fuzzy subset composed of the test environmental factors is:

$$\begin{aligned} C_1 &= [0.598, 0.345, 0.058] \\ C_2 &= [0.408, 0.333, 0.266] \\ C_3 &= [0.332, 0.341, 0.327] \end{aligned}$$

According to the principle of maximum membership degree, the influence degree of each factor is respectively:  $C_1 = 0.598, C_2 = 0.408, C_3 = 0.341$  ( $C_1 > C_2 > C_3$ ). It is shown that the influence degree of 3 environmental factors to the permeability tests hydrogenated nitrile O-ring is temperature, pressure and volume fraction of CO<sub>2</sub> from high to low.

#### 2.6. Analysis of interaction between two factors

Interaction is refers to the influence degree of various environmental factors between each other and the combination of maximum influence value can be calculated through the fuzzy matrix between factors. Environmental factors have 3 levels and there are 9 collocation ways between interaction factors. Taking the interaction  $C_1$  and  $C_2$  as an example, the fuzzy matrix between them is:

$$\begin{aligned} C_1^T \times C_2 &= \begin{bmatrix} (\sum b_{11})' \\ (\sum b_{12})' \\ (\sum b_{13})' \end{bmatrix} \begin{bmatrix} (\sum b_{21})' & (\sum b_{22})' & (\sum b_{23})' \end{bmatrix} \\ &= \begin{bmatrix} (\sum b_{11})'(\sum b_{21})' & (\sum b_{11})'(\sum b_{22})' & (\sum b_{11})'(\sum b_{23})' \\ (\sum b_{12})'(\sum b_{21})' & (\sum b_{12})'(\sum b_{22})' & (\sum b_{12})'(\sum b_{23})' \\ (\sum b_{13})'(\sum b_{21})' & (\sum b_{13})'(\sum b_{22})' & (\sum b_{13})'(\sum b_{23})' \end{bmatrix} = \begin{bmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{bmatrix} = A_{lk} \end{aligned} \quad (7)$$

**Table 4**

The processing results of fuzzy comprehensive evaluation.

	Level	Temperature factor	Pressure factor	Volume(CO <sub>2</sub> ) factor
The total of fuzzy comprehensive evaluation	$\sum b_{11}$	2.640	1.790	1.465
	$\sum b_{12}$	1.521	1.460	1.506
	$\sum b_{13}$	0.254	1.165	1.444
Membership grade	$(\sum b_{11})'$	0.598	0.408	0.332
	$(\sum b_{12})'$	0.345	0.333	0.341
	$(\sum b_{13})'$	0.058	0.266	0.327

$A_{jk}$  is the fuzzy relation on the domain of  $X_i \times X_j$ . The elements represent the combined effects of the match between two different levels to the fuzzy comprehensive evaluation in the formula.

The following result can be got by taking the data in Table 3 into Formula (7).

$$C_1^T \times C_2 = \begin{bmatrix} 0.598 \\ 0.345 \\ 0.058 \end{bmatrix} \begin{bmatrix} 0.408 & 0.333 & 0.266 \end{bmatrix} \\ = \begin{bmatrix} 0.244 & 0.199 & 0.159 \\ 0.141 & 0.115 & 0.092 \\ 0.024 & 0.019 & 0.015 \end{bmatrix} = R_{12}$$

Taking the principle of maximum membership degree to determine the best combination with only considering the combination between two factors. If only the combination between  $C_1$  and  $C_2$  taken into consideration, the best combination is  $C_{11}$  and  $C_{21}$ . In the same way, the other membership grade of best combination between two factors is  $C_{11}$  and  $C_{31}$ ;  $C_{21}$  and  $C_{31}$ . Therefore, the interaction of temperature and pressure is maximum.

### 3. Conclusions

- (1) The self-designed device is used to simulate the downhole environment of oil and gas exploitation, which work well in permeability test of rubber O-ring. Taking the orthogonal experiment method and fuzzy orthogonal analysis method can greatly reduce the work and has the advantages of convenience and accuracy to find the main environmental factor and interaction effect, which provides a new way to study the influence of downhole complex environment on the performance of rubber sealing material.
- (2) According to the results of fuzzy orthogonal analysis, the influence degree of every environmental factor on permeability test of hydrogenated nitrile O-ring from large to small is temperature, pressure and concentration of CO<sub>2</sub> under the limit work conditions. Besides, the interaction of

temperature and pressure are the most significant to the comprehensive index. Therefore, temperature and pressure are major considerations environmental factors to effect the sealing performance of hydrogenated nitrile O-ring and need to avoid at the high value for the sealing failure will become more severe. The result have important reference value to further study the failure mechanism of rubber sealing ring in many environmental factors and the rational use in engineering.

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